



Standard Test Method for Pour Point of Petroleum Products (Automatic Pressure Pulsing Method)¹

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INTRODUCTION

This test method covers an alternative procedure for the determination of pour point of petroleum products using an automatic apparatus.

1. Scope*

1.1 This test method covers the determination of pour point of petroleum products by an automatic instrument that applies a controlled burst of nitrogen gas onto the specimen surface while the specimen is being cooled and detects movement of the surface of the test specimen with an optical device.

1.2 This test method is designed to cover the range of temperatures from -57°C to $+51^{\circ}\text{C}$. However, the range of temperatures included in the 1992 interlaboratory test program only covered the temperature range from -39°C to $+6^{\circ}\text{C}$ and the range of temperatures included in the 1998 interlaboratory test program was from -51°C to -11°C . (see 13.4).

1.3 Test results from this test method can be determined at 1°C or 3°C testing intervals.

1.4 This test method is not intended for use with crude oils.

NOTE 1—The applicability of this test method or residual fuel samples has not been verified. For further information on applicability, refer to 13.4.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*²

- D97 Test Method for Pour Point of Petroleum Products
- D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
- D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
- D6708 Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material

2.2 *Energy Institute Standard:*

- IP 15 Test Method for Pour Point of Petroleum Products³

3. Terminology

3.1 *Definitions:*

3.1.1 *pour point, n—in petroleum products*, the lowest temperature at which movement of the test specimen is observed under the prescribed conditions of the test.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *no-flow point, n—in petroleum products*, the temperature of the test specimen at which a wax crystal structure or viscosity increase, or both, impedes movement of the surface of the test specimen under the conditions of the test.

3.2.1.1 *Discussion*—The no-flow point occurs when, upon cooling, the formation of wax crystal structures or viscosity increase, or both, has progressed to the point where the applied observation device no longer detects movement under the

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ Available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K., <http://www.energyinst.org.uk>.

conditions of the test. The preceding observation temperature, at which flow of the test specimen is last observed, is the pour point.

3.2.2 *pulse, n*—a controlled burst of nitrogen gas of a fixed pressure and flow rate sufficient to cause movement on the surface of the test specimen without fracturing the wax structure which may be formed in the specimen.

3.2.3 *Peltier device, n*—a solid-state thermoelectric device constructed with dissimilar semiconductor materials, configured in such a way that it will transport heat to or away from a test specimen dependent on the direction of electric current applied to the device.

4. Summary of Test Method

4.1 After inserting the test specimen into the automatic pour point apparatus, and initiation of the test program, the test specimen is heated and then cooled by a Peltier device at a rate of $1.5\text{ }^{\circ}\text{C}/\text{min} \pm 0.1\text{ }^{\circ}\text{C}/\text{min}$. At temperature intervals of $1\text{ }^{\circ}\text{C}$ or $3\text{ }^{\circ}\text{C}$, depending on the selection made by the user, a moving force in the form of a pressurized pulse of compressed gas is imparted onto the surface of the specimen. Multiple optical detectors are used in conjunction with a light source to monitor movement of the surface of the specimen. The lowest temperature at which movement of the specimen surface is observed upon application of a pulse of compressed gas is recorded as the pour point, Test Method D5949.

5. Significance and Use

5.1 The pour point of a petroleum product is an index of the lowest temperature of its utility for certain applications. Flow characteristics, like pour point, can be critical for the correct operation of lubricating oil systems, fuel systems, and pipeline operations.

5.2 Petroleum blending operations require precise measurement of the pour point.

5.3 In most cases, this test method does not require the use of mechanical refrigeration apparatus (see 7.1).

5.4 This test method yields a pour point in a format similar to Test Method D97/IP 15 when the $3\text{ }^{\circ}\text{C}$ interval results are reported.

NOTE 2—Since some users may wish to report their results in a format similar to Test Method D97 (in $3\text{ }^{\circ}\text{C}$ intervals) the precisions were derived from the temperatures rounded to the 3° intervals. For statements on bias relative to Test Method D97, see 13.3.

5.5 Test results from this test method can be determined at either $1\text{ }^{\circ}\text{C}$ or $3\text{ }^{\circ}\text{C}$ intervals.

5.6 This test method has better repeatability and reproducibility relative to Test Method D97/IP 15 as measured in the 1992 and 1998 interlaboratory test programs.⁴

⁴ The results of this interlaboratory test program are available from ASTM International Headquarters in the form of a research report. Request RR:D02-1312 for the 1992 program and RR:D02-1499 for the 1998 program.

6. Apparatus

6.1 *Automatic Apparatus*⁵—The automatic pour point apparatus described in this test method consists of a microprocessor controlled test chamber that is capable of heating and cooling the test specimen, providing a controlled pulse of compressed gas onto the specimen surface, optically detecting the specimen surface movement, and recording the temperature of the specimen as described in detail in Annex A1. It is specifically designed to detect the lowest temperature at which movement of the surface of the specimen is observed upon application of the pulse.

6.2 The apparatus shall be equipped with a specimen cup, an array of optical detectors, light source, pressure pulsing unit, digital display, Peltier device, and a specimen temperature measuring device.

6.3 The pressure pulsing unit consists of a stainless steel tubing, $250\text{ mm} \pm 2\text{ mm}$ long and $1.1\text{ mm} \pm 0.1\text{ mm}$ inside diameter. This tubing is connected to a constant pressure source at one end, which serves as an inlet. The other end of the tubing, which serves as the outlet, is bent and positioned such that it is pointing to the center of the specimen at an acute angle. The distance between the outlet and the center of the specimen is $8\text{ mm} \pm 2\text{ mm}$.

6.4 The Peltier device shall be capable of heating or cooling the test specimen at a rate of $1.5\text{ }^{\circ}\text{C}/\text{min} \pm 0.1\text{ }^{\circ}\text{C}/\text{min}$.

6.5 The temperature measuring device in the specimen cup shall be capable of measuring the temperature of the test specimen from $-80\text{ }^{\circ}\text{C}$ to $+70\text{ }^{\circ}\text{C}$ at a resolution of $0.1\text{ }^{\circ}\text{C}$.

6.6 The apparatus, if required,⁵ shall be equipped with fittings to permit the circulation of water or other liquid cooling media to remove heat generated by the Peltier device and other electronic components of the apparatus. Newer models⁵ have internal sources of liquid cooling media and do not require such fittings.

6.7 The apparatus shall be equipped with fittings to permit the delivery of nitrogen gas to the pressure pulsing unit. Newer models⁵ have internal sources of compressed gas and do not require such fittings.

6.8 *Ultrasonic Bath, Unheated*—(optional)—with an operating frequency between 25 kHz to 60 kHz and a typical power output of $\leq 100\text{ W}$, of suitable dimensions to hold container(s)

⁵ The following instrument has been found suitable for use in this test method: Phase Technology Pour Point Analyzer model series 30, 50, 70, 70V and 70X; available from Phase Technology, 11168 Hammersmith Gate, Richmond, B.C. Canada V7A 5H8. In the 1998 research report, the 70V was referred to as the *current* model; whereas models 30, 50, and 70 were referred to as *pre-1998* models. The various model series mentioned above are differentiated by their cooling capacities and user interfaces; however, all of them are capable of covering the entire temperature range specified in the scope.

Model series 30, 50, 70, and 70V require external sources of coolant and compressed gas (dry nitrogen). Model series 70X has built-in internal sources of coolant and compressed gas.

This pour point analyzer is covered by a patent. Interested parties are invited to submit information regarding the identification of an alternative to this patented item to the ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee which you may attend.